IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of	
DEME, Imants	
Serial No. 10/582,060	Group Art Unit: 1793
Filed: June 8, 2006	Examiner: Pegah Parvini
SULPHUR PELLET COMPRISING H2S-SUPPRESSANT	

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER RULE 37 CFR 1.132

Imants Deme hereby declares:

THAT I received a BSc. in Civil Engineering in 1964 from the University of Manitoba, Winnipeg, Manitoba, Canada; and that I received a MSc. Degree in Engineering in 1969 from the University of Manitoba, Winnipeg, Manitoba, Canada; and

THAT I was employed by the Manitoba Highway Department, Materials and Research Division, Winnipeg, Manitoba, Canada from 1964 to 1969 and by Shell Canada Limited and its related companies in Canada, United Kingdom, and Australia from 1970 to 2001; and

THAT I have been the Principal of the consulting company Asphalt Services Inc., Burlington, Ontario, Canada since 2001 and have performed consulting services for Shell Canada Limited and the Saudi Arabian American Oil Company and have worked in the areas of asphalt technology and sulfur-asphalt technology with applications principally in roads and roofing and various other related applications; and

THAT I performed a study and obtained the following Additional experimental results as described below.

Additional experimental results

Experimental Procedure.

The ingredients used in the study were as follows.

1. Aggregate: Dolomitic limestone + siliceous blending sand (Aggregate gradation:100% pass 19 mm; 55% pass 4.75 mm; 2.6% pass 0.075 mm), Nelson Aggregates, Burlington, Ontario.
2. Bitumen: 150/200 penetration grade, Husky Energy, Lloydminster, Alberta.
3. Sulphur: Sampled from a molten sulphur delivery truck and allowed to solidify; from Bay Sulfur Company, Stockton, California. H_2S content is unknown, but it is known that this sulphur prilling plant receives sulphur from various sources ranging in H_2S contents from 10 ppm to over 100 ppm.
4. Ferric Chloride Solution (28 – 43%): Eaglebrook, Inc. of Canada, Varennes, Quebec.

The $FeCl_3$ content selected for the study was 0.5% solids based on sulphur weight. As the $FeCl_3$ solution was approximately 35% solids, 1.43% of the aqueous solution was used.

Commercially manufactured sulphur pellets or prills were not available from the same sulphur feedstock as the $FeCl_3$ modified sulphur, essential for proper comparison. The pellets were therefore simulated for the lab study by using crushed sulphur slate. They were produced by first melting the supplied sulphur and then pouring it in approximately a 4 mm thickness onto aluminum foil placed on a flat surface. After cooling the slate was crushed to form particles up to 5 mm size to simulate sulphur pellet size.

Similarly, the simulated $FeCl_3$-modified sulphur "pellets" were prepared by melting the elemental sulphur, mixing with aqueous $FeCl_3$ until all of the water had evaporated, casting it into slate form on a flat surface and then breaking it up into fine particles after cooling.

The sulphur-asphalt paving mixture composition was 93.8% aggregate, 3.7% bitumen and 2.5% sulphur product. The sulphur-asphalt mixture was prepared in 2500 gram batches.

The aggregate, bitumen, mixing bowl and scraper were all heated in an oven to 160 °C for the blends made with untreated sulphur-asphalt mix and the sulphur modified with 0.5% ferric chloride solids. This temperature for the ingredients was selected from experience to compensate for the temperature loss during mixing to a mix target temperature near 145 °C, the target for H_2S measurement. The oven temperature was raised to 170 °C for the sulphur-asphalt

mix incorporating 1.43% aqueous $FeCl_3$ (NB: yields 0.5% $FeCl_3$ solids) to compensate for the additional heat loss from driving off the water, in order to yield the same target sulphur-asphalt mix temperature for fume emission tests, near 145 °C, as for the other sulphur-asphalt mixes. (NB: 140 ±5 °C is the usual target mixing temperature for sulphur-asphalt paving mixtures at commercial asphalt hot-mix plants and 145 °C, at the upper limit of the range, is the target temperature for scavenger effectiveness studies.)

The sulphur-asphalt mix was prepared by first adding hot bitumen to the hot aggregate and then mixing for 30 seconds. The sulphur, sulphur and H_2S scavenger/inhibitor, and sulphur incorporating H_2S scavenger/inhibitor were then added respectively for the three mix types at room temperature and mixing was continued for 90 seconds. The sulphur-asphalt mix was then transferred to an insulated four-litre vessel, which half-filled it. A loose cover was placed on the vessel to allow H_2S gas to accumulate in the air space above the mix. The H_2S concentration was measured after five minutes using a Drager MiniWarn gas meter equipped with a pump and a suction hose, drawing the gas through a hole in the cover from the air space above the mix at the rate of 20 l/min. Mix temperature was measured before and after the gas test, with the mean value quoted in Table 1.

Observations.

The test results, summarized in Table 1 below, indicated that the H_2S concentration in the air space above the mix had accumulated to a level of 54 ppm after 5 minutes when commercial grade sulphur was used to make the sulphur-asphalt paving mixture. Addition of 0.5% $FeCl_3$ solids reduced the H_2S concentration to 15 ppm. However, the H_2S level was reduced further to 6 ppm when sulphur was used which already had 0.5% $FeCl_3$ solids incorporated.

Table 1:

Effect of Scavenger Addition Method on H_2S Evolution from Sulphur-Asphalt Paving Mixtures (measured immediately after mixture preparation). The H_2S concentration in the air space above the mix was measured after 5 minutes.

Product(s) added to hot paving mix	**Temperature paving mix (°C)**	**H_2S concentration (ppm)**
Commercial grade sulphur (no additives)	141	54
Sulphur + 0.5 % $FeCl_3$ added separately	146	15
Sulphur pellet containing 0.5 % $FeCl_3$	143	6

Conclusions.

The lab study confirmed that H_2S scavengers and inhibitors, intimately combined with sulphur in pellets, can both reduce the H_2S emissions associated with the use of gassier sulphurs as well as retarding and/or reducing the H_2S emissions attributed to the reaction between sulphur and bitumen in hot sulphur-asphalt paving mixtures. This is a big difference, compared to the same amount of scavenger/inhibitor added to the mix generally, and a surprising result that was not expected.

Imants Deme further declares that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: July 8, 2008

Imants Deme

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